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AD 681206

CCL REPORT NO. 258

FINAL REPORT

DETERMINATION OF THE NONVOLATILE CONTENT OF COATINGS
IN PRESSURIZED AEROSOL SPRAY CONTAINERS

BY

C. W. CROSS

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M. H. SWANN

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U. S. ARMY COATING & CHEMICAL LABORATORY

Aberdeen Proving Ground
Maryland

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AMCMS CODE NO. 5025.11.29500

DEPARTMENT OF THE ARMY PROJECT NO.

1T062105A329

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ABSTRACT

Construction of simple sampling devices is illustrated for quantitatively removing small samples of material from aerosol spray cans and a technique is described for the rapid determination of nonvolatile content. A satisfactory method for this purpose has not been previously available.

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I. INTRODUCTION

There is no detailed procedure available that is satisfactory for the determination of the nonvolatile content of enamels, varnishes or lacquers in pressurized aerosol containers. Precise measurement of percent nonvolatile composition of any coating material is perhaps the most important single analytical test to be made. In the case of coatings that are sprayed from pressurized cans, the proportion of such ingredients as solvent, pigment, resin and propellant are critically important for satisfactory performance. While there are numerous analytical procedures for all types of coating materials in general, they are practically nonexistent for the packaged aerosol coating products. The identification of the propellants through the use of gas-liquid chromatography was described by Esposito (1, 2) in which the eight propellants that can be recommended for use with coating materials were clearly separated and identified. The only method given in detail for measuring the nonvolatile content appears in Federal Specification TT-L-50d (3). This procedure requires the destruction of an entire container of spray material and is so time-consuming that the analysis can seldom be completed the same day it is started. In addition, there is some doubt that the method is accurate since the results rarely agree with the manufacturer's stated contents. Briefly, the method in TT-L-50d involves chilling the weighed container in a refrigeration unit to such a low temperature that the pressure is removed, puncturing the can and pouring out the contents, then rinsing with solvent until all of the coating has been quantitatively removed. The empty can must be reweighed and the contents allowed to warm to room temperature. A water bath is then used to reduce the contents to less than 50% of their original weight, followed by re-dilution to an exact fraction of the original amount. The nonvolatile is then obtained on this portion and corrected to the "whole contents" basis.

Several methods were proposed in an article by Sciarra (4) for obtaining the approximate composition of these products but most of the procedures are not sufficiently detailed to exhibit practicality or have overlooked the extreme difficulty of quantitatively collecting the finely divided particles dispersed from a spray nozzle in a suitable container. Some of the proposals made by Sciarra are not suitable for pigmented materials. His recommended procedure for routine use consists of measuring the density with a hydrometer of the pre-chilled contents at -20°F. and calculating the nonvolatile from this figure. The main deficiency of this technique is the varying density of the various propellants and mixtures of propellants that may be encountered.

The technique developed in this laboratory for determining the percent nonvolatile of the contents of pressurized spray cans, as described in this report, involves construction of a sampling device from common laboratory materials, which provides for quantitative removal of 1 to 2 grams of sample from a weighed container of paint. The small sample is then rinsed from the sampling device into a suitable container, dried and weighed. There is no need for chilling of the sample to reduce pressure

and the method is non-destructive so that the remainder of the contents are unaffected and may be used. The time required for completion of a test is only slightly greater than for other standard nonvolatile methods. Some typical analyses of lacquers and enamels are shown in Table 1 (Appendix A).

II. DETAILS OF TEST

A. Reagent - Acetone or benzene, technical grades, appropriate to the type of coating.

B. Apparatus

Laboratory balance, 750 or 1500 gram capacity, with sensitivity to 2 milligrams. Fisher Scientific catalog item 1-967, 1-968 or equivalent.

Beakers, 250 ml. capacity.

Oven, 105°C.

Shaker, reciprocal motion, Fisher "Equipose" or equivalent, Fisher catalog item 14-261 (this item not absolutely necessary as hand-shaking of the aerosol containers can be substituted).

Sampling Device, see Figures 1 and 2 (Appendix B).

C. Construction of sample transfer devices - Removal of the spray nozzle from a pressurized can will reveal the type of sampling device needed; a recessed outlet will require the type illustrated in Figure 1, while a protruding outlet will necessitate the type shown in Figure 2. In both cases, a 100-ml. bottle made of "Nalgene" is perforated with about 5 small holes by means of a heated needle, to serve as a pressure release. These perforations are located at the top edge of the shoulder of the bottles as shown in the illustrations. In the first of these two types, a release valve is prepared by cutting away the excess plastic from the center tube of a spray nozzle taken from a spray can. This release tube is connected to plastic tubing with a clamp and the plastic tubing is connected to glass tubing by pre-heating in hot water. The Protruding clamp also serves as a point of support for applying pressure during sampling.

In the second type of sampler (Figure 2), an additional single perforation is made in the bottom center of the plastic bottle with a heated needle, in such a manner as to fit snugly around a 1/16-inch metal rod that extends all the way through the device. This rod serves to depress the recessed valve of the spray can by exerting pressure on the opposite end. The 4 mm. glass tubing is prepared to fit snugly around protruding outlet of the can and a perfect fit can usually be obtained by gently fire-polishing the outer end of the glass tubing while periodically cooling and checking for size. At the time of sampling, both devices are used in an inverted position from that shown in the drawings.

D. Procedure - It is advisable to remove the paper labels and clean the sides of the cans to be sampled; the label can be replaced at the conclusion of the test, if desired. Remove the spray nozzle from the sample can. Weigh the can as accurately as the high-capacity balance will allow and shake for 15 minutes, preferably in a reciprocal-motion type of agitator. As soon as possible after agitation, remove 1 to 2 grams with the sampling device in the following manner:

Type I (recessed outlet) - Support the can at a 45 degree angle, insert the valve tip of the sample device (Figure 1), making sure that the perforated vent holes of the bottle are in a topside position, then press down quickly and firmly for approximately one second.

Type II (protruding outlet) - Support the pressure can at a 45 degree angle, position the glass tube over the outlet, turn the bottle so that the vent holes are topside, hold down firmly and press down on the 1/16-inch metal rod for one or two seconds.

Disengage the sampling device and rinse the contents into a weighed beaker with a suitable solvent. If a small amount of paint spills onto the sample can, rinse that portion also into the beaker. Evaporate the solvent in a water bath under a gentle stream of air to low volume then transfer to an oven at 105°C. for one hour. Cool the beaker in a desiccator and weigh. Re-weigh the aerosol can and calculate the percent nonvolatile.

$$\% \text{ Nonvolatile} = \frac{\text{Gain in weight of the beaker} \times 100}{\text{Loss in weight from the sample can}}$$

III. RESULTS AND DISCUSSION

The sampling devices may also be used for collecting larger samples from pressurized containers for such purposes as pigment determinations, isolation of vehicle or pigment analyses.

Samples of fully known composition were not available. Values obtained were compared to manufacturer's stated composition with fairly good agreement in most cases. When lower values were obtained, the samples were subjected to excessive agitation and repeated analyses on the assumption that yields would increase if error was due to insufficient shaking. Repeatability was poorest in the case of metallic pigments such as "copper bronze".

IV. REFERENCES

1. Coating and Chemical Laboratory Report No. 226, "Identification of Aerosol Propellants in Paint Products by Gas-Liquid Chromatography," March 1967.

2. Esposito, G. G., J. Paint Tech. 39, 338 (1967).

3. Specification TT-L-50d, "Lacquer, Nitrocellulose and Acrylic, Aerosol, Halogenated Hydrocarbon in Pressurized Cans, November 1967.

4. Sciarra, J. J., Paint & Varnish Production, 55, 67 (1965).

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Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1 ORIGINATING ACTIVITY (Corporate author) U. S. Army Coating and Chemical Laboratory Aberdeen Proving Ground, Maryland 21005		2a REPORT SECURITY CLASSIFICATION Unclassified	
		2b GROUP	
3 REPORT TITLE DETERMINATION OF THE NONVOLATILE CONTENT OF COATINGS IN PRESSURIZED AEROSOL SPRAY CONTAINERS			
4 DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Report			
5 AUTHOR(S) (Last name, first name, initial) Cross, C. W., and Swann, M. H.			
6 REPORT DATE January 1969		7a TOTAL NO OF PAGES 14	7b NO OF REFS 4
8a CONTRACT OR GRANT NO AMCMS Code No. 5025.11.29500		9a ORIGINATOR'S REPORT NUMBER(S) CCL No. 258	
b PROJECT NO IT062105A329		9b OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
c			
d			
10 AVAILABILITY LIMITATION NOTICES Qualified requestors may obtain copies of this report from Defense Documentation Center. Distribution of this document is unlimited.			
11 SUPPLEMENTARY NOTES		12 SPONSORING MILITARY ACTIVITY U. S. Army Materiel Command Washington, D. C.	
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KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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